New furnace for high temperature GISAXS experiments at LNLS

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Objective

Allow for simultaneous GISAXS (*Grazing Incidence Small Angle X-ray Scattering*) and XRD (*X-ray Diffraction*) experiments with controlled temperature and environment for *in situ* kinetic studies of nanoparticles growth and quenching processes.

Introduction

A new system has been proposed for different GISAXS experiments at LNLS, allowing temperatures as high as 1000 ˚C and pressures down to 10^{-7} mbar, as well as inert gases. Intending to simultaneously perform XRD experiments, a special design was developed for the furnace window, which is presently made of Kapton, but might be upgraded to beryllium at some point. A prechamber is also included so that transient effects might be very quickly studied if a sample would be transported to the furnace already at the appropriate temperature. The heater with a removable sample holder was customized from Heatwave Labs. Finally, within the given budget, the best way to have all the necessary degrees of freedom for alignment was to use a Huber 4 + 2 diffractometer at one LNLS' beamlines. Therefore, only one extra motor has been added to the system to adjust the sample height inside the chamber. Limited in load and space by the diffractometer, the final design can be seen in figures 1 and 2.

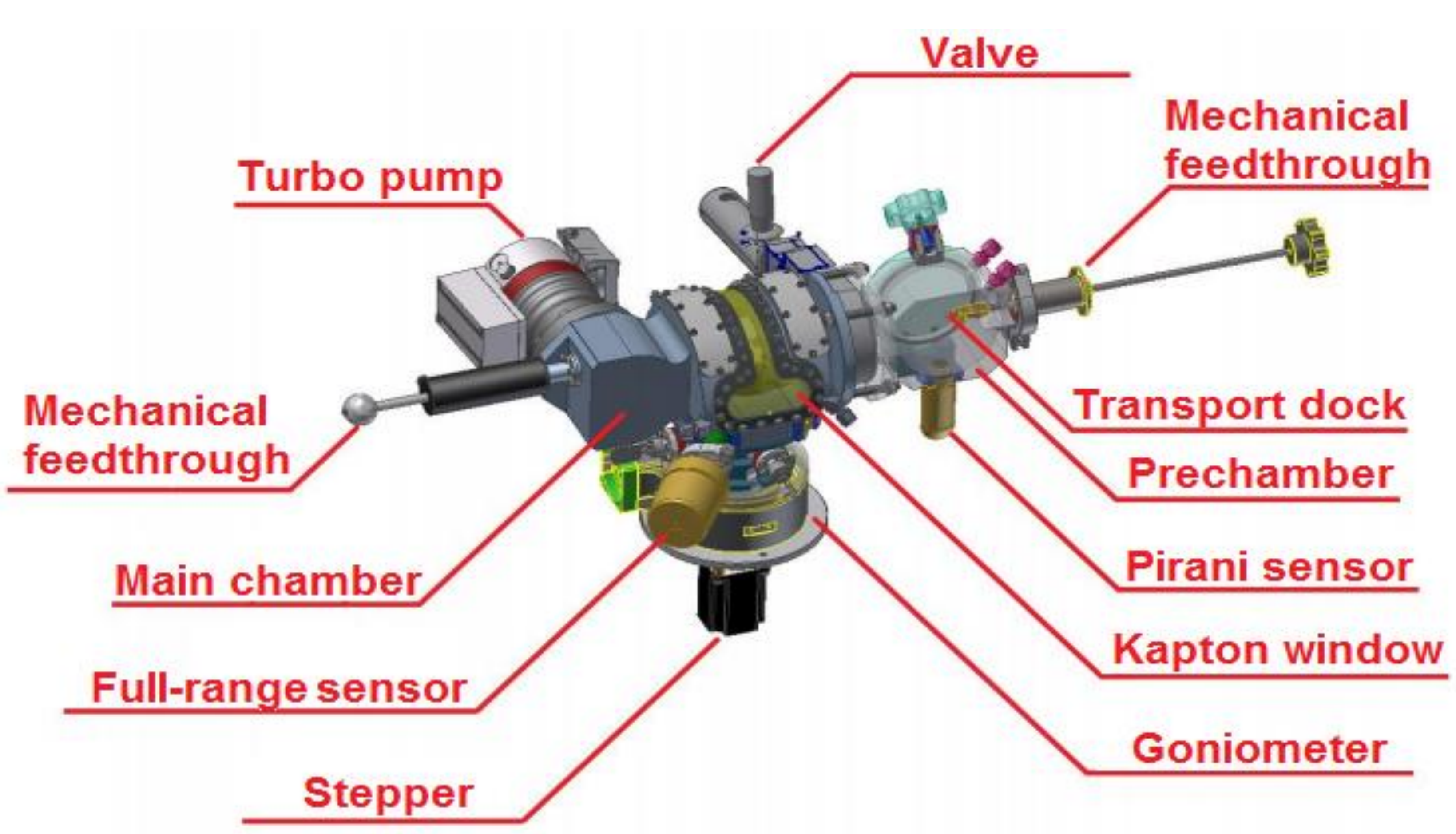

Fig. 1: Perspective view of the furnace with its components.

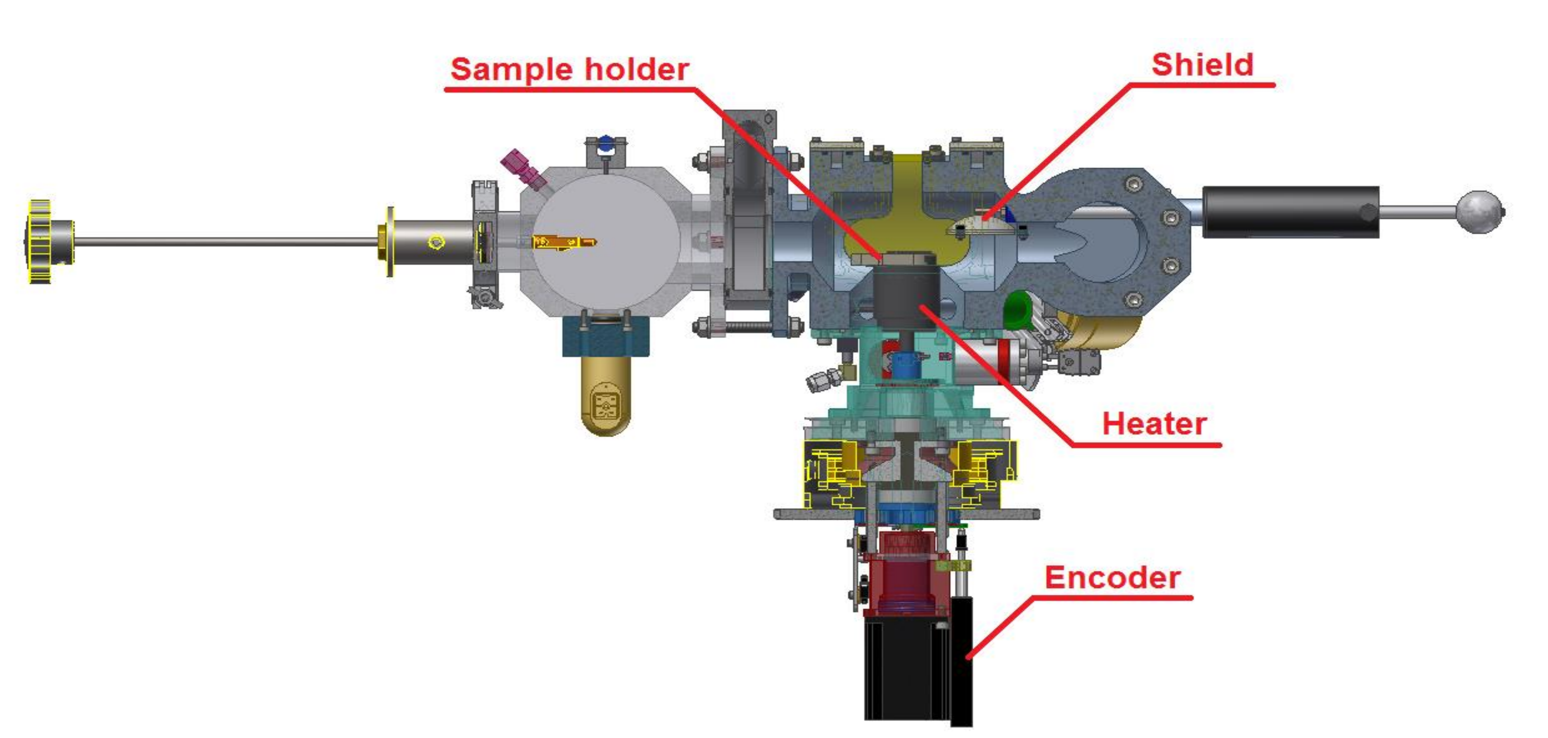

Fig. 2: Section view of the furnace, showing the heating element, the removable sample holder, the shield and the encoder.

Results

Preliminary bench tests revealed that a base pressure of 5×10^{-5} mbar may be successfully achieved within a couple of minutes, what is enough for the moment with the Kapton window. In addition, the cooling systems for both the heater and the chamber have been validated, and the shield has proved itself to be unnecessary, since the window temperature stabilizes at about 150 ˚C. On the other hand, although the heater did reach 1200 ˚C, the heating concept unfortunately prevented the sample from reaching more than 800 ˚C. Nonetheless, the sample holder coupling and the leveling system have also performed well.

Before the system is tested by users at XPD beamline in December 2014, it was subject to a diffraction experiment with a MgO standard powder sample in October, as seen in figure 3. These results are presently under analysis.

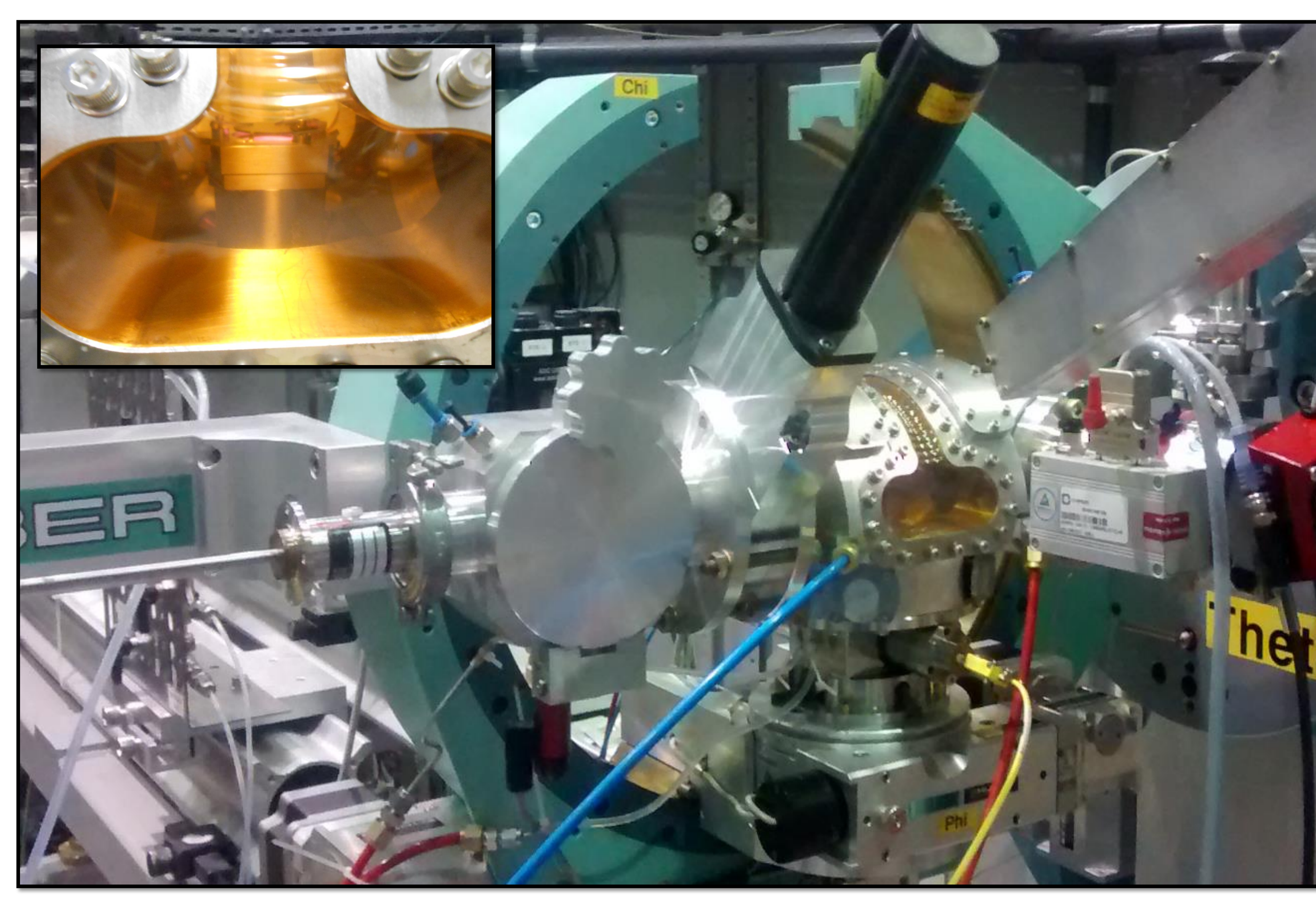

Fig. 3: Setup on a Huber 4+2 diffractometer at XPD beamline. The insert shows the heater and the sample holder during the experiment.

Conclusions

The system is likely sufficient for a first round of experiments by the community of users. Even though, a few improvements may be already mentioned, as: replacement of the Kapton window by a beryllium window for better vacuum levels; automation of the valve and the mechanical feedthrough for better transient analyses; and rework on the heating concept and design for higher temperatures.

Acknowledgements

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